ORIGINAL ARTICLE

The effect of surface conditioning methods on shear bond strength of resin luting cement to all ceramic coping material. An in – vitro study

RKV PATEL, ABHINAV AGARWAL, KALAVATHY N.

Abstract

This study was conducted to evaluate the effect of surface conditioning methods on shear bond strength of resin luting cement to All – Ceramic material. Four different ceramic surface treatments were tested: silane, sandblasting + silane [Group 2], 9%HF acid + silane [Group 3], and sandblasting + 9%HF acid + silane [Group 4]. 10 All - Ceramic samples were fabricated with each surface treatment and bonded using dual cure composite resin luting cement. The total number of samples amounted to 40. All the 40 samples were subject to shear bond strength tests and observed for the type of bond failure. The force required to debond the samples was recorded and the shear bond strength was calculated. Group-3 demonstrated greatest mean bond strength followed by Group- 4, Group- 2 and then Group- 1. Group 1 and Group 2 Samples produced predominantly Adhesive failures; Group 3 and Group 4 samples produced predominantly cohesive failures both inside the luting cement and porcelain and cohesive failures only inside the porcelain. The use of surface conditioning methods improved the shear bond strength between the composite resin luting cement and ceramic. HF acid etching + silane produced the highest bond strength and hence this was seen as the method of choice for bonding composite resin luting cement to Glass ceramics.

Key words: Ceramic surface conditioning, shear bond strength, bond failure


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Introduction:

All – Ceramic crowns have been in use for more than 70 years. The past decade witnessed a growth in the use of all ceramic materials because of their excellent esthetic qualities, structural durability, chemical inertness, wear resistance and good biocompatibility when compared to metal ceramic restorations. Even though the combination of predictable strength and reasonable esthetics has continued to make traditional metal ceramic restorations popular, patient demand for improved esthetics has driven the development of All - Ceramics for use with inlays, onlays, crowns, FPD’s, post and cores and implant supported restorations¹. Glass ceramics have become one of the most widely used categories of all ceramic materials because they are the most translucent appearing ceramic materials available¹. However clinicians are still facing problems related to fracture of ceramic restoration because of their brittleness and limited flexural strength³ hence definitive adhesive cementation with composite resin should be used to increase the fracture resistance of the restorations¹.
Resin bonding of ceramic restoration to the supporting tooth structure increases retention, marginal adaptation, and fracture resistance of the restored tooth and the restoration. Adhesive cementation improves the fracture resistance of the ceramic material by penetrating the flaws and irregularities on the internal surface of the restoration and by inhibiting crack propagation. Previous investigations have revealed that most clinical failures have initiated from the resin cement and ceramic interface since most adhesive resin luting agents have adequate dentine and enamel bonding properties and poor bonding properties to unconditioned ceramic. Therefore the integrity of the luting cement to the ceramic surface plays a major role in the longevity of the restoration and the failures originated from the cementation surfaces identified the need for a reliable conditioning method to strengthen this critical area.

To create a reliable bond between the ceramic material and adhesive resin different surface treatment methods for ceramics have been employed which mechanically facilitate the bonding. They are grinding or abrasion with diamond rotary instruments, air borne particle abrasion with aluminum oxide, acid etching with acidulated phosphate fluoride gel and hydrofluoric acid and tribo-chemical silica coating. The resin ceramic bonding can also be enhanced by application of silane coupling agent to the ceramic surface prior to bonding with resin cement. Silane coupling agents usually contain a silane coupler and a weak acid which enhances the formation of siloxane bonds to produce chemical bonding. They also increase the wettability of the ceramic surface.

Since the use of All Ceramic restoration require considerable support from the underlying composite luting cement and enamel/dentine, this study was conducted with the aim of evaluating the shear bond strength and type of bond failure that can occur between resin luting cement and all ceramic material.

The objectives of the study were:
1. To compare and evaluate the shear bond strength of All – Ceramic coping material to resin luting cement between 4 different surface treatment group’s.
2. To evaluate the type of bond failure which can occur with each of the surface treatment tested.

Materials and Methods:
A machined was prepared. Using Inlay Wax, wax rods of 5.5mm diameter were fabricated from the cylindrical stainless steel die with 5.5mm diameter hole and 20mm height (Figure 1).

![Figure 1 - Cylindrical stainless steel die and wax rods fabricated from the stainless steel die.](image-url)
ceramic rods of 5.5mm diameter and 20mm length (Figure 2).

(Figure 2 - Ceramic rods)

The ceramic rods were cut into discs of 3mm thickness using diamond cutting disc and their thickness was evaluated with the caliper (Figure 3).

(Figure 3 – Ceramic discs evaluated for thickness with caliper.

80 such ceramic discs were prepared which were made flat by abrading with wet 600 grit silicon carbide paper and were cleaned in ultrasonic bath containing distilled water for 5min and air dried.

40 ceramic discs were randomly selected and embedded in 40 stainless steel molds containing self cure acrylic resin ensuring the top surface of the ceramic disc remained uncovered for the bonding procedure and the remaining 40 free ceramic discs were used later for bonding to the embedded ceramic discs.

Grouping of samples –
The 40 embedded ceramic samples and the 40 free ceramic samples were randomly divided into 4 groups such that each group will contain 10 free ceramic samples and 10 embedded ceramic samples.(figure 4)

(Figure 4 - Grouped ceramic samples.

Each group of ceramic specimens received different surface treatments which were as follows –

**Group 1 ( Control )** – No surface treatment was performed. Silane solution was applied over the ceramic samples with applicator tip and allowed to air dry for 5minutes.

**Group 2** – The ceramic specimens were air abraded with 110micrometer aluminum oxide at 2.5 bar pressure for 20 seconds. All specimens were then rinsed under running tap water for 60 sec to remove aluminum oxide particles and air dried. This was followed by application of silane solution with applicator tip and allowed to air dry for 5min.

**Group 3** – Ceramic specimens were etched with 9% HF acid for 3min and rinsed in running tap water for 60 sec to remove residual acid after etching and air dried. This was followed by application of silane solution with applicator tip and allowed to air dry for 5min.
**Group 4** – Ceramic samples were air abraded with 110 micro meter aluminum oxide at 2.5 bar pressure for 20 sec. All specimens were rinsed under running tap water for 60 sec to remove aluminum oxide particles and air dried. This was followed by group 3 surface treatment.

**Ceramic cement bonding procedure** –
A piece of adhesive polyethylene tape with a 4.5 mm diameter circular hole was fixed on the center of the embedded ceramic specimen surface to define the area of the bond and to maintain 50 micrometer thickness of luting cement.

The desired equal lengths of base and catalyst pastes were dispensed onto the mixing pad and mixed according to manufacturer’s instructions. The mixed cement was applied to the exposed surface of embedded ceramic disc using plastic mixing spatula.

The Free ceramic samples and embedded ceramic samples of the same group (Identically treated) were bonded together with resin luting cement. Thus we get 10 specimens from each group.

The designed sample holder secured the specimens in a rigid position under the load of 750 gm during the bonding procedure. Excess cement was removed with the explorer tip before complete hardening of resin luting cement and then photo polymerized for 60 sec from two opposite directions using LED curing unit. All the 40 bonded specimens were immersed in distilled water and incubated at 37°C for 24 hrs.

The shear load was applied to the specimens until failure in a universal testing machine at cross head speed of 0.5 mm/min and the load that caused failure was recorded for each specimen.

**Evaluation of bond failure** - After shear testing, de bonded surfaces of all specimens were observed through an stereo microscope at 40X magnification to assess the mode of bond failure and the failures were recorded as –

- A- Adhesive failure at the luting cement porcelain interface (Figure 5).

![Figure 5 - Type A bond failure](image)

B – Combination of cohesive and adhesive failures with occasional crack propagation inside the porcelain (Figure 6)

![Figure 6 - Type B bond failure](image)

C – Cohesive failures both inside the luting cement and porcelain (Figure 7).
The mean shear bond strength values for all the four different groups are summarised in table I (graph 1).

Graph 1 - Comparison of mean shear bond strength for the four different groups
Group-3 demonstrated greatest mean bond strength of (20.52 ± 2.67) followed by Group-4 (17.74 ± 1.89), Group-2 (10.52 ± 1.62) and Group-1 [Control] which demonstrated the lowest bond strength (2.44 ± 0.60). Table II presents factorial ANOVA results for the shear bond strength and the analysis revealed that all the three test groups (Group-2, Group-3, and Group-4) were significantly different from the Control /Group-1 (p = < 0.001).

Post Hoc analysis – Fisher’s LSD [Table III] revealed that the shear bond strength of Group 2 (10.52 ± 1.62) is statistically significant from Group-3 (20.52 ± 2.67) and Group-4 (17.74 ± 1.89) (p = < 0.001), where as there was no significant difference between Group-3 (20.52 ± 2.67) and Group-4 (17.74 ± 1.89) samples.

Table IV shows the different types of failures in all the four groups (graph 2).

Graph 2 - Comparison of different types of failures for the four different groups
Type A failure was predominant in Group-1 [10 (100%)] and Group-2 [8 (80%)] samples, Type B failures was very low and was found in Group-2 [2 (20%)] and Group-4 [1 (10%)] samples, Type C failures was predominantly found in Group-3 [7 (70%)] and Group-4
[7 (70%)] samples and Type D failures was also very low and was found in Group-3 [3 (30%)] and Group-4 [2 (20%)] samples. Kruskal-Wallis Rank test [Table V] showed that Group-3 samples with the mean rank (31.35) presented the better bonding followed by Group-4 (29.55), Group-2 (11.6) and Group-1 (9.5) respectively. The failure mode analysis results also strongly supported the results of shear bond strength and showed poor bonding of Control samples, unstable bonding of Group-2 samples and stronger bonding of the Group-3 and Group-4 samples.

Table I – Mean Shear bond strength (Mpa) and statistical analysis results:

<table>
<thead>
<tr>
<th>Statistics</th>
<th>N</th>
<th>Mean</th>
<th>Standard Deviation</th>
<th>Range</th>
<th>95% Confidence interval for mean</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Lower bound</td>
</tr>
<tr>
<td>Group 1</td>
<td>10</td>
<td>2.44</td>
<td>0.603</td>
<td>(1.839-3.509)</td>
<td>1.08</td>
</tr>
<tr>
<td>Group 2</td>
<td>10</td>
<td>10.52</td>
<td>1.629</td>
<td>(8.459-12.96)</td>
<td>6.83</td>
</tr>
<tr>
<td>Group 3</td>
<td>10</td>
<td>20.52</td>
<td>2.672</td>
<td>(15.926-25.39)</td>
<td>14.48</td>
</tr>
<tr>
<td>Group 4</td>
<td>10</td>
<td>17.74</td>
<td>1.893</td>
<td>(14.446-20.169)</td>
<td>13.46</td>
</tr>
</tbody>
</table>

Table II - ANOVA for 2 X 2 Factorial Experiments:

<table>
<thead>
<tr>
<th>Source of Variation</th>
<th>Degrees of freedom</th>
<th>Sum of squares</th>
<th>Mean Sum of squares</th>
<th>F ratio</th>
<th>p value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group 2</td>
<td>1</td>
<td>70.08</td>
<td>70.08</td>
<td>20.40</td>
<td>0.00007</td>
</tr>
<tr>
<td>Group 3</td>
<td>1</td>
<td>1601.74</td>
<td>1601.74</td>
<td>466.17</td>
<td>0.00000</td>
</tr>
<tr>
<td>Group 4</td>
<td>1</td>
<td>294.65</td>
<td>294.65</td>
<td>85.76</td>
<td>0.00000</td>
</tr>
<tr>
<td>Error component</td>
<td>36</td>
<td>123.69</td>
<td>3.44</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>39</td>
<td>2090.17</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table III - Post hoc Analysis – Fisher’s LSD.

<table>
<thead>
<tr>
<th>Pair</th>
<th>p value</th>
<th>Interpretation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group 2 vs. Group 3</td>
<td>0.0062</td>
<td>Significant</td>
</tr>
<tr>
<td>Group 2 vs. Group 4</td>
<td>0.0425</td>
<td>Significant</td>
</tr>
<tr>
<td>Group 3 vs. Group 4</td>
<td>0.4236</td>
<td>Not significant</td>
</tr>
</tbody>
</table>

Table IV – Mode of failures observed after shear testing:

<table>
<thead>
<tr>
<th>Failure mode/Score</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>TOTAL</th>
</tr>
</thead>
<tbody>
<tr>
<td>A/1</td>
<td>10 (100%)</td>
<td>8 (80%)</td>
<td>-</td>
<td>-</td>
<td>18 (45%)</td>
</tr>
<tr>
<td>B/2</td>
<td>-</td>
<td>2 (20%)</td>
<td>-</td>
<td>1 (10%)</td>
<td>3 (7.5%)</td>
</tr>
<tr>
<td>C/3</td>
<td>-</td>
<td>-</td>
<td>7 (70%)</td>
<td>7 (70%)</td>
<td>14 (35%)</td>
</tr>
<tr>
<td>D/4</td>
<td>-</td>
<td>-</td>
<td>3 (30%)</td>
<td>2 (20%)</td>
<td>5 (12.5%)</td>
</tr>
<tr>
<td>TOTAL</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>40 (100%)</td>
</tr>
</tbody>
</table>
Table VI: Post hoc analysis – Mann Whitney U test

<table>
<thead>
<tr>
<th>Pair</th>
<th>p value</th>
<th>Interpretation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group 2 vs. Group 3</td>
<td>&lt; 0.025</td>
<td>Significant</td>
</tr>
<tr>
<td>Group 2 vs. Group 4</td>
<td>&lt; 0.025</td>
<td>Significant</td>
</tr>
<tr>
<td>Group 3 vs. Group 4</td>
<td>&lt; 0.025</td>
<td>Significant</td>
</tr>
</tbody>
</table>

Discussion:

Bond strengths between ceramic and tooth are influenced by several factors one of which is the type of luting cement. A variety of cements and bonding techniques have been applied to modern All – Ceramic restorations. Zinc phosphate, zinc polycarboxylate and conventional GIC set through an acid base reaction and have a tendency to exacerbate surface flaws in ceramic restorations. Due to the increased acidity of the cement GIC is susceptible to early water degradation, resulting in micro cracks which may initiate cracks and facilitate crack propagation in the cement. Considering the brittleness and limited flexural strength of glass ceramics, definitive adhesive cementation with composite resin should be used to increase the fracture resistance of the restoration. In addition, these resin cements which are available in multiple shades are tailored to ceramic restorations and enable exact shade matching and subtle shade corrections to be made.

To create a reliable bond between the ceramic and adhesive resin, different surface treatments have been employed which mechanically and chemically facilitate the bonding. These surface treatment methods increase the surface area to facilitate mechanical
interlocking thereby enhancing ceramic composite adhesion.

Another important factor for obtaining a sufficient resin bond to silica based ceramics is the application of silane coupling agents \(^7, ^{12}\).

In this study ceramic to ceramic bonded specimens were used for evaluating the single effect of the Ceramic – Resin interface on the bond strength. This system is capable of eliminating other factors such as resin to dentin bond strength, which may affect the recorded ceramic to resin bond strength.

This study suggested that the shear bond strength of ceramic to composite is affected by ceramic surface treatments. 9% HF acid etching for 3min followed by silane application proved to be the most effective and reliable surface treatment \(^{13, ^{14}}\). The bond strength resulting from this method was significantly different (\(p < 0.001\)) from only silane treatment and sandblasting followed by silane treatment, however it was not significantly different from HF + sandblasting + silane.

It can be hypothesized that 9% HF acid applied for 1-4 min selectively dissolves the glass matrix and produces a porous irregular surface which increases the surface area and facilitate the penetration of resin materials into the micro undercuts of the etched porcelain surface \(^6\) and also produces superior adaptation of cements to ceramics \(^{15}\). Whereas sandblasting with 110µm alumina only removed significant amount of substances from the ceramic surface, weakening the ceramic and failed to effectively produce the micro undercuts to increase the surface area and the bond strength \(^{16}\).

The silane solutions are composed of bi-functional molecules that bond silicon dioxide with the –OH groups [Hydrogen bonds] on the ceramic surface and usually contains a silane coupler and a weak acid, which enhances the formation of siloxane bonds \(^7\).

They also have a degradable functional group that copolymerizes with the organic matrix of the resin. Several authors have previously shown that silanization also increases the wet ability of the ceramic surfaces \(^2\).

This study also suggested that there is no significant difference in bond strength values between HF acid etching + silane treatment and combination of sandblasting + HF acid etching + silane treatment, and since sandblasting restorations with 110 µm alumina (\(\text{Al}_2\text{O}_3\)), has the potential to remove significant amounts of substances and could weaken the prostheses, hence unnecessary sandblasting should be avoided \(^{14}\).

The quality of the bond strength should not be assessed based on bond strength data alone, the mode of failure should also provide important information leading to prediction of clinical performance limits.

Adhesive failure at the luting cement porcelain interface occurred in 100% of control samples and 80% of Group 2 samples. Group 3 samples showed cohesive failures both inside the luting cement and porcelain (70%) and cohesive failures inside the porcelain (30%). Group 4 specimens showed cohesive failures both inside the luting cement and porcelain (70%), cohesive failures inside the porcelain (20%) and Combination of cohesive and adhesive failures with occasional crack propagation inside the porcelain type failures (10%).

These findings corresponds with the observed bond strengths, showing that micromechanical interlocking plays an important role in resin – ceramic bonding. This failure mode analysis
results supported the results of shear bond strengths and indicated poor bonding of controls, unstable bonding of sandblasted + silanized samples and strong bonding of the HF acid etching + silane samples and sandblasting + HF acid etching + silane treated samples.

Conclusion:
Within the limitations of this in vitro study, the following conclusions were drawn:
1. The use of surface conditioning methods improved the shear bond strength between the composite resin luting cement and ceramic when compared with the untreated group.
2. HF acid etching + silane produced the highest bond strength followed by HF + sandblasting + Silane, sandblasting + silane and silane [control group] respectively.
3. Samples prepared using silane [control] and sandblasting + silane showed adhesive failures indicating poor bonding.
4. Samples prepared using HF acid + silane and HF + sandblasting + Silane showed the cohesive failures within the porcelain and both inside the luting cement and porcelain indicating stronger bonding.
5. The use of 9% HF acid followed by silane application is the method of choice for bonding composite resin luting cement to Glass ceramics.

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